

MACRO AND MICRO RESIDUAL STRESS DISTRIBUTION IN 6061Al-15 vol.% SiC_w UNDER DIFFERENT HEAT TREATMENT CONDITIONS

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ABSTRACT

The residual stress, RS, profiles in a 6061Al-15vol.%SiC_w composite and **in unreinforced 6061Al alloy** in ~~its alloy matrix~~ have been determined by synchrotron radiation diffraction, SRD. The high spatial resolution achieved by this technique has allowed resolving the spatial dependence of all three principal components of the RS field with the sample radius. The micro and macro stress have been successfully separated revealing the different distribution along the sample cross section as a consequence of their different nature. In this way clear experimental evidence of theoretical works could be given.

KEYWORDS: Discontinuously Reinforced Metal Matrix Composites (DRMMCs); 6061Al; Micro and Macro-Residual Stress. Synchrotron radiation, heat treatment.

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INTRODUCTION

Discontinuously reinforced metal matrix composites, DRMMCs, such as Al alloys reinforced by SiC, are being increasingly used in structural applications because of their enhanced mechanical properties with respect to the corresponding unreinforced alloys. These composites have the advantage that they can be re-processed via conventional metallurgical procedures such as extrusion, rolling, forging, and machining to obtain the final component shape. Consequently, their reasonable manufacture costs and superior mechanical properties allow them to compete with monolithic metallic alloys. These properties are strongly

dependent on the microstructure and on the presence of a residual stress, RS, state in the material [1]. RS heavily affects the life in service [2] as it superimpose to applied stress.

The RS state of DRMMCs is more complex than that in the unreinforced alloys because of the presence of both macroscopic-RS, M-RS, and microscopic-RS, m-RS, as defined in [3]. The M-RS is caused by the thermo-mechanical processes that take place during the material fabrication and ulterior heat treatments. According to [4], the m-RS is due to two main sources: i) the thermal mismatch and ii) the elastic mismatch between matrix and reinforcement. In the absence of plastic pre-straining and M-RS, the average microscopic (thermal) RS is compressive in the reinforcement and tensile in the matrix, fulfilling the stress balance on the grain size scale, and therefore on the whole material. In this case, the m-RS is supposed to be independent of the position in a component. On the contrary, the M-RS distributes along the specimen (or component) differently, depending on its geometry and on the thermo-mechanical process involved. Moreover, the M-RS must be balanced on the whole sample volume or along any section [5]. The knowledge of the distribution of the RS is important to identify failure modes and “hot spots”, where cracks are likely to form. Some theoretical work has been done (see e.g. [6]) to assess the M-RS spatial distribution in simple cases, and the problem is generally considered already solved in textbooks [7]. As a consequence, only few non-destructive experimental confirmations (mainly by means of neutron diffraction, ND) are available. For example, Fitzpatrick *et al.* [4] studied the profile around a crack tip of a 2124 Al matrix composite. They reported the distribution of stress along the crack growth direction and its variation with applied load. Dutta *et al.* [8] investigated the variation of the RS as a function of plastic deformation in Al alloy matrix composite bars subjected to four-point-bending tests. They could resolve the typical W profile and found that also the m-RS is space-dependent (i.e. it depends on the plastic deformation). This confirmed previous work by Levy-Tubiana *et al.* [9], who added the plastic deformation of the matrix to the possible sources of m-RS. However, further experimental confirmations are still needed to validate the theoretical calculations. For example Fitzpatrick *et al.* [10] just *assume* a parabolic profile for the RS state of a quenched 2124Al-17%vol.SiC_p composite bar. Their data shows some scatter. This is partly due to the limitations of the spatial resolution achievable with ND.

Medium and high energy (40-150 keV) Synchrotron Radiation Diffraction, SRD, has proved to be a very powerful tool to investigate RS in materials and industrial components [11]. In particular, the spatial resolution achievable is often superior to other techniques. Therefore, if no particular coarse grain problems occur, as in the present case, SRD is ideal to tackle the problem of profiling RS in samples and/or components with a stress spatial range of variation

of the order of 1 mm. As it will be seen later, the problem of the elongated gauge volume, which often hinders 3-D stress analysis, can be solved with a suitable experimental set-up and careful theoretical considerations.

In this work a SRD experiment has been carried out in order to outline the RS profiles along the diameter of cylindrical samples of 6061Al 15%vol SiC_w composite and unreinforced 6061Al alloy. These profiles have been studied after different heat treatment conditions (i.e. in different precipitation states), in order to understand the influence that regular heat treatments applied to this class of alloy matrix composites have also on their initial RS developed.

EXPERIMENTAL DETAILS

Materials and Samples

The samples used in this study were cylinders (6.5 mm diameter, 13 mm length) of 6061Al alloy and 6061Al-15%vol.SiC_w composite, both produced by powder metallurgy [12]. The composite was labelled E219 and the matrix E220. The cylinders were machined from bars extruded at about 500°C through a flat die with an extrusion ratio of 27:1, which implied 3.30 true strain. This severe reduction led to a highly textured matrix material ($\langle 111 \rangle$ and $\langle 001 \rangle$ fibre texture components, with the fibre axis parallel to the extrusion axis) and to some trend of the SiC_w (with an average aspect ratio of about 2) to be aligned with the extrusion axis. Details about the processing route, the texture, and the microstructure of these materials are given in [12,13]. The RS profiles were studied after three precipitation states, T4 or “as-quenched”, T6 or fully hardened condition and OA or over-annealed. The T4 condition was obtained after a solution treatment at 520°C for 90 min, followed by quenching in cold water. The T6 condition was achieved by successive annealing at 146°C during 56h for the matrix and 16h for the composite, as the precipitation kinetics is accelerated by the addition of the reinforcement [13]. The OA condition was achieved by further annealing at 300°C for 100 h, for both the alloy and the composite.

Experimental

The SRD experiments were carried out on the beamline ID31, at the ESRF, Grenoble, France. This beamline has a particular advantage: being equipped with an analyser crystal, it is virtually insensitive to partial immersion of the gauge volume [14]. The beam dimensions chosen were 80 μm height and 500 μm width. This ensured that in every sample tilt position a spatial resolution of about 500 μm could be guaranteed (see below and appendix). The energy was 60 keV, which corresponds to a wavelength $\lambda = 0.207 \text{ \AA}$. The 311 planes for both the Al and the SiC phases were measured. They correspond to $2\theta \sim 9.7^\circ$, and 9.05° , respectively. The

resulting gauge volume is a rhomboid prism of $0.08 \times 0.94 \times 0.5 \text{ mm}^3$ (short diagonal, long diagonal and width, respectively). It can be shown that 50% of the long diagonal identifies 75% of the diffracting volume (see appendix). So, in spite of the gauge volume elongation (Fig.1.d), we could assume that the spatial resolution was $500\mu\text{m}$ in all 3 directions.

The $\sin^2\psi$ method [15] was used: the sample was tilted within the scattering plane between $\psi = 0^\circ$ (axial direction) and $\psi = \pm 90^\circ$ (radial or hoop direction). The ψ angle is that between the scattering vector and the extrusion axis, see Fig.1. The sample holder was made of polymethylmethacrylate (PMMA) to minimise beam absorption. The samples were mounted on the sample holder as shown in Fig.1. The tilt range available on ID31 was not large enough to cover the whole ψ interval. For this reason, two different setups were used; with the samples mounted vertical and horizontal (see Figs.1.b and c).

A cylindrical co-ordinate system (axial, radial and hoop axes) has been adopted, due to the symmetry of the extrusion process. The principal stress directions coincide with the sample geometrical ones. In order to obtain the radial and hoop strain components, measurements were performed along radii in both Z and X directions, see Fig.2. If the extrusion axis is parallel to the scattering vector \mathbf{q} , the axial strain component is measured. When the extrusion axis is perpendicular to \mathbf{q} , the radial component is measured in a scan along X (X scan), and the hoop in a scan along Z (Z scan), as shown in Fig.2. The hoop and radial strain components were not measured at the very same point, but rather at two equivalent points located on perpendicular diameters at the same radius. The equivalence of the stress state along a given circumference is valid under the reasonable assumption of a cylindrical symmetry. The pitch of the scans was 0.49 mm in the unreinforced alloy and 0.59 mm in the composite. The minimum distance between the last point and the sample surface was 0.3 mm . In this manner, 13 points in E220 and 11 in E219, including the sample centre in both cases, were measured. The distribution of the measurement points and the orientation of the samples during the measurements of the axial and radial strain components are shown in Fig.2.

RESIDUAL STRESS ANALYSIS

From the SRD measurements the values of lattice spacing, d , were obtained. By comparing d with the stress-free value, d_0 , the strain at each location within the sample is calculated from:

$$\varepsilon = \frac{d - d_0}{d_0} \quad (1)$$

The strain values in the principal directions were obtained by a linear fit of the ε vs. $\sin^2 \psi$ plots [15] recorded at each point. These plots allow mapping the strain ellipsoid in the axial/radial and in the axial/hoop planes at each location in the sample.

The *total* stress corresponds to the measured strains and was therefore simply calculated by means of the Hooke's law [16].

$$\sigma_i^T = \frac{E}{(1+\nu)(1-\nu)} [(1-\nu)\varepsilon_i + \nu(\varepsilon_j + \varepsilon_k)] \quad (2)$$

where i, j , and k are the principal directions (axial, radial and hoop upon permutation of indices), E is Young's modulus and ν Poisson's ratio.

In the composite E219, the macro stress (M-RS) σ^M was calculated from the phase-specific total stress $\sigma_{Al/SiC}^T$ by means of the rule of mixtures (ROM), which reads:

$$\sigma^M = (1-f)\sigma_{Al}^T + f\sigma_{SiC}^T \quad (3)$$

f is the reinforcement volume fraction. The micro stress (m-RS) $\sigma_{Al/SiC}^m$ could then be calculated by means of:

$$\sigma_{Al/SiC}^m = \sigma_{Al/SiC}^T - \sigma^M \quad (4)$$

The balance of the axial macro RS (M-RS) component σ_{ax}^M across the sample was used for the determination of d_o [5,17]. In cylindrical geometry, this condition reads:

$$\int_{-R}^R \sigma_{ax}^M \cdot r dr = 0 \quad (5)$$

where R is the sample radius. Since a finite number of measurements has been made, equation (5) can be rewritten as:

$$\sum_i \sigma_{ax,i}^M |r_i| \Delta r_i = 0 \quad (6)$$

where r_i are the measurement positions and Δr are the gauge volume projections along r . Upon inserting eqs.(1)-(3) in equation (6) (i.e. expressing it as a function of the interplanar spacings d and d_0), one can calculate d_0 using the measured values of $d_{(ax,rad,hoop),i}$, r_i , and Δr at each point i :

$$d_0 = \frac{(1-f) \sum_i \left[A \cdot d_{i,ax}^{Al} + B \cdot (d_{i,rad}^{Al} + d_{i,hoop}^{Al}) \right] \cdot |r_i| \cdot \Delta r_i}{(1-f) \cdot (A + 2 \cdot B) \sum_i |r_i| \cdot \Delta r_i - f \sum_i \sigma_{i,ax}^{T,SiC} \cdot |r_i| \cdot \Delta r_i} \quad (7)$$

In eq.(7) $A = E(1-\nu)/[(1+\nu)(1-2\nu)]$ and $B = E\nu/[(1+\nu)(1-2\nu)]$. This procedure was used for each material in each condition. Obviously, in the case of the unreinforced alloy $\sigma_{Al/SiC}^T = 0$ and $f = 0$. The diffraction elastic constants for the 311 Al and SiC peaks were calculated using a Kröner model [18]:

$$\begin{aligned} E_{311}^{Al} &= 69 \text{ GPa} & \nu_{311}^{Al} &= 0.33 \\ E_{311}^{SiC} &= 370 \text{ GPa} & \nu_{311}^{SiC} &= 0.19 \end{aligned}$$

RESULTS

Figure 3 shows some examples of the ε vs. $\sin^2\psi$ plots, as determined in the centre of samples E220 and E219 (in the OA and T4 conditions). At each location in the sample ε depends linearly on $\sin^2\psi$. This implies that the strong fibre texture does not heavily influence the stress distribution (as a function of the tilt angle ψ). The fact that no ψ -splitting was observed confirms that the geometrical sample directions are indeed principal [15].

Stresses were calculated by means of the above-mentioned procedure. In the following, the main findings are reported.

a) Macroscopic RS

Figure 4 shows the M-RS components (axial, radial and hoop) along the sample radius for the composite and the unreinforced matrix case. The RS states shown correspond to T4, T6, and OA conditions (Fig.4.a,b,c respectively). All stress components in the T4 and T6 conditions show parabolic profiles (the fitting curves are also shown), although the radial stress profiles look flatter than the others. In the OA condition the radial and hoop components have a flat distribution, so they were fitted with a constant.

The maximum (tensile) stress σ_{\max} lies at the sample centre, whereas the minimum (compressive) stress σ_{\min} lies at the surface. The axial component shows the largest absolute stress and the largest variations. For any given heat treatment, the axial and hoop stress variations are larger in the alloy than in the composite. Expectedly, the samples have some axial macro deviatoric character due to the sample geometry [16].

Samples in the T4 condition show the largest stress variation along the radius. In T4 the surface compressive hoop stress is larger than the radial analogous, in E220 more than in E219. Sample E219 shows larger error bars than E220, because of the poorer signal detected on the SiC phase. The error of macro-RS was also calculated from standard error propagation theory [19], through the ROM.

The parabolic profile flattens considerably after the T6 treatment. The axial stress in the composite E219 and in the unreinforced alloy E220 is essentially the same. The hoop and radial components differ in the two samples only by a constant.

After the OA treatment the profile of all stress components is almost completely flat and near to zero in both the alloy E220 and the composite E219: the stress has basically relaxed. It must be noticed that in all precipitation conditions for E219 and in the over aged for E220 there is a constant shift between the axial component profile and both radial and hoop profiles. This rigid translation along the stress axis is around 40 MPa in E219 and 15 MPa in E220 OA. This is most probably due to an instrumental effect, which will be discussed below.

b) Microscopic RS

The m-RS profiles of the composite E219 have been also calculated; they are shown in Fig.5. The m-RS is tensile in the matrix and compressive in the reinforcement, as expected. Contrarily to the M-RS, the m-RS is constant as a function of radius.

The m-RS values for the SiC phase show larger dispersion and error bars. No particular curve shape is visible, and it can be assumed that each component is constant as a function of radius. All heat treatment conditions show similar m-RS values: around 60 MPa (± 15 MPa) for the matrix and -350 (± 100) MPa for the reinforcement. Therefore, no evolution of the micro-RS could be observed with the heat treatment. Besides, the values of the three principal components are very similar, since the differences fall within the error bars. This reveals that the micro RS is essentially hydrostatic.

c) Peak width

The peak width (as obtained from a Gaussian fit of the diffraction peaks) at each radial position has been obtained as the average of all peak widths measured at different ψ tilt angles. The matrix peak width shows the most significant features and will be represented for sake of clarity. Figure 6 shows the variation of the Al-311 peak width with the sample radius in the composite and the unreinforced alloy in the three heat treatment conditions.

No dependence was found of the peak width from the ellipsoid branch investigated (i.e. the two different set-ups used, see above). In other words the peak width in the axial, radial and hoop direction is basically identical, as it is to be expected. Therefore the average between all measurements at equivalent radii was taken.

DISCUSSION

From a physical point of view it is impossible to have a fully relaxed axial stress while still having non-zero (and constant) radial and hoop stresses (see Fig.4). It is therefore reasonable to state that in the OA condition, the radial and hoop M-RS should also vanish. The observed displacement between axial and radial/hoop stresses must then be due to a systematic instrumental error. Most probably, the use of two different setups has led to an energy shift caused by a re-alignment of the monochromator. Since this alignment procedure is automatic, this instrumental error cannot be easily estimated. However, since its effect is a rigid displacement of the profiles the conclusions of this work are not affected by this systematic error.

a) Variation of macroscopic RS

In order to rule out the effect of the systematic shift of the hoop and radial stress profiles, it is instructive to compare the total range of variation of all M-RS components (Table 1). This is taken as the stress difference between those at the centre and at the surface of the samples. In fact, the relaxation of the M-RS is visible in Fig.4, but can be readily quantified from Table 1. Reduction factors from the T4 to the T6 conditions of about 4 for the radial and axial stress components and about 8 for the hoop component can be calculated.

The relaxation factor from the T6 to the OA condition is again of the order 4 (for E220) to 10 (for E219) for the axial stress (Table 1), whereas the other two components (Fig.4c) relax completely and therefore present no variation. Because of this, the deviatoric stress is also slightly parabolic after annealing (with a total range of variation of 15 MPa).

It has been mentioned above that the rapid cooling after quenching (to reach the T4 condition) causes a parabolic temperature gradient in the sample [20]. Consequently, all macro stress components show a parabolic behaviour as a function of the radial position (Fig.4). Some further considerations on the RS nature and evolution with heat treatments can be done separating the hydrostatic and axial deviatoric stresses. These stresses read respectively:

$$\sigma_{Hyd} = \frac{\sigma_{Ax} + \sigma_{Rad} + \sigma_{Hoop}}{3} \quad (8)$$

$$\sigma_{Dev} = \sigma_{Ax} - \sigma_{Hyd} \quad (9)$$

Fig.7 shows that both the deviatoric and the hydrostatic stresses have parabolic profiles in the unreinforced alloy E220 for the T4 and the T6 state (Fig.7.a). On the other hand, in sample E219 the axial deviatoric component is essentially constant (Fig.7.b). In the T6 conditions (in

both samples) all profiles are sensibly flatter than in T4 (Figs. 4 and 7.b). This indicates that annealing at 146°C partially relaxes the M-RS. This is in disagreement with the results found out in previous works on high strength aluminium alloys [21] and with previous interpretation of neutron diffraction measurements proposed by the authors [22]. The present results indicate that annealing at 146°C indeed favours activity of the dislocation density associated to the precipitation (annihilation and/or rearrangement).

In the unreinforced alloy E220 the axial stress dominates since the deviatoric stress profile follows the axial. This result can be explained as follows: when quenching leads to plastic flow due to the severe temperature gradient, plastic flow occurs preferentially along the axial direction as dictated by the boundary conditions: Along the cylinder axis the dilation/contraction is unconstrained, while it is along the hoop and radial directions (periodic boundary conditions and smaller size, respectively). This is confirmed also by the profiles of the hydrostatic stress: they are similar to the axial stress profile in every case. On the other hand, in E219 the reinforcement has a damping effect on the matrix plastic flow introducing further local boundary conditions. Therefore, the strain tends to redistribute and to become hydrostatic. The fact that the deviatoric stress is almost constant in the composite E219 means that the profiles of all components have the same curvature. This implies that the radial temperature gradient has the same effect on each stress component. In fact, the observed non-zero values of the deviatoric M-RS (about 30 MPa) could even be attributed to the above-mentioned systematic error and it could be reckoned that the M-RS is essentially hydrostatic in both the T4 and the T6 conditions.

In Fig.8 the hydrostatic and deviatoric RS profile relaxation from the T6 to the OA condition is apparent by using a different y -scale. As in Fig.7, both profiles are parabolic in sample E220 in the T6 conditions, but relax significantly after the annealing treatment at 300°C.

The same happens for the composite E219 (Fig.8b). The hydrostatic M-RS has ranges of variation of 30 MPa in T6 and essentially 0 in the OA condition. This suggests a complete relaxation. This observation disagrees with previous work [21] and therefore further work is needed to determine the influence of d_0 and of the systematic error found above.

For the composite E219, the profile of the deviatoric M-RS is found to be basically flat in the OA condition, as seen for T4 and T6. Its value is then independent of the heat treatment. This supports the idea that in E219 the M-RS is again essentially hydrostatic due to the constrained plastic flow and that the finite value is only an artefact due to the mentioned systematic error. The fact that the deviatoric stress relaxes with the annealing treatment in the unreinforced alloy and remains constant in the composite is in full agreement with previous investigation carried out by neutron diffraction [22].

b) Variation of microscopic RS

The three main components of the m-RS in the composite are summarised in Table 2. For the matrix, they correspond to the average of the three central points (which would correspond to the same gauge volume used in the neutron diffraction experiments of [21]). In the reinforcement, it is taken the average of all points taken along the diameter, due to the larger data scatter:

The deviatoric and the hydrostatic stress profiles are shown in Figure 9. Contrarily to the M-RS, the m-RS is constant along the radius, which points out their different origin. As it has been mentioned above, the m-RS results from the thermal mismatch (i.e. the difference of the thermal expansion coefficients, CTEs) between the matrix and the reinforcement. Therefore, it is the same at every point along the sample diameter. The deviatoric term is also constant and near to zero. This stress isotropy is somewhat unexpected because of the fibre-shape of the reinforcement. Indeed, neutron diffraction measurements [22] showed some small deviatoric m-RS in E219 (about 15 ± 10 MPa). On the other hand, it should be taken into account that the aspect ratio of the whiskers is small (around 2) and a basic absence of strength differential effect was found in this material [23]. Both latter data strongly support the findings of the present work.

Another important point is that the three precipitation (i.e. heat treatment) conditions studied present the same values of m-RS. Previous works have, instead, pointed out some relaxation of micro residual stress [22]. In [22], the m-RS has been measured in the centre of the sample by means of neutron diffraction, i.e. with a larger gauge volume. For the sake of comparison with refs. [22,23], the three central values of m-RS from SRD are taken in Table 2. They show that the possible m-RS variation, not only from T6 to OA, but also from T4 to T6, is of the order of the error bars. Moreover, if the magnitude of the m-RS after the quenching from 520°C (T4) is the same as that achieved after a slow cooling from 300°C (OA), one can conclude that the micro RS is independent from one of the most important heat treatment parameters, the cooling rate. This seems also to indicate that the Eshelby equivalent temperature [19] lies also below 300°C. At high temperature the GNDs probably move and annihilate relaxing the m-RS, but it seems that they always re-generate on cooling leading to the same level of m-RS.

c) Variation of the Peak Width

It has been reported that the increase in lattice distortion due to the presence of dislocations leads to an increase in the peak width [24]. This is fully confirmed in the present work, because in the unreinforced alloy the peak width is less than the half of that observed in the composite. In the latter, there is indeed a much higher density of dislocations because of the presence of GNDs.

In the unreinforced alloy in the T4 condition, the peak width profile shows that the distortion near the surface is higher than that at the sample centre. This is an expected result, as the major thermal shock during the quenching takes place at the surface. In the T6 condition, the distortion relaxes more at the surface regions as the dislocations here move during the heat treatment.

In the composite, there is not such a clear peak width gradient after the quenching step. Nevertheless, the peak width (lattice distortion) tends to reduce with the annealing for the T6 condition in the whole sample, Fig. 6. This reduction, not detected in the unreinforced alloy, is probably due to local rearrangements of the GNDs. These dislocations may have developed high energy configurations which can be partially released with the annealing for the T6 condition. This does not contradict the observed fact that the microstructure of these composites is more stable than those of the unreinforced alloys [25]. Finally, as in the case of the unreinforced alloy E220, the activity of dislocations in E219 seems to be higher at the surface: The profile of the peak width flattens completely after overaging.

CONCLUSIONS

The residual stress (RS) distribution within the section of cylindrical samples of a 6061Al 15%vol. SiC_w composite and its unreinforced matrix has been investigated by means of synchrotron radiation diffraction. The RS evolution with different heat treatments has been observed. The following points summarize the main results of this study:

a) Stress balance considerations:

Highly spatially resolved measurements along the radius allowed the use of the axial macro stress equilibrium equation within the sample section. From this equation, the reference lattice parameter d_0 were calculated in each heat treatment condition.

b) Separation of phase specific stresses:

From the diffraction data, phase-specific stresses could directly be determined for the Al and the SiC phase. Using the ROM also a separation of the macro (common to both phases) and the micro (phase specific) stress could be done.

c) Macro-RS evolution

The distribution of the three main components of the Macro-RS (axial, radial and hoop) was found to be parabolic, with the sample centre in tension stress and the edges under compression. The following features were detected:

- In the unreinforced alloy, after the T4 treatment the axial component is larger than the other two because of the bigger effect of the temperature gradients: the plastic flow is favoured in the axial direction and limited in the hoop and radial directions, because of geometrical constraints. This effect is not present in the composite, where the reinforcement favours stress re-distribution. In fact, the axial stress is larger in the unreinforced matrix than in the composite. This can also be due to the larger CTE of the former.
- The parabolic profiles flatten with heat treatments: The residual stress present in the T4 condition (as quenched) relaxes significantly after annealing for the T6 condition. In spite of a systematic instrumental error, with further annealing (to the OA condition) the stress seems to relax completely in the composite, but not in the unreinforced alloy, in which some (small) axial stress remains.
- A finite axial deviatoric macro stress is present after the extrusion process. In the unreinforced alloy, this stress is only reduced after the ageing process from T4 to T6, and with further annealing from T6 to OA. On the other hand, it looks to be essentially relaxed in the composite E219, where a hydrostatic stress state seems to be present.

d) Micro RS Considerations:

The micro RS profiles are essentially constant along the sample diameter, tensile in the matrix and compressive in the reinforcement. The most remarkable features are:

- The hydrostatic stress does not vary with the heat treatment in both the matrix and the reinforcement. This shows the different nature of micro and macro RS, and supports the idea that micro RS arises systematically as a consequence of the interface mismatch between matrix and reinforcement.
- The deviatoric component is also constant with the heat treatment and around zero. This fact points out the isotropy in the micro residual stress.

- The diffraction peak width, related to the lattice distortion, is higher in the composite, due to the higher dislocation density provided by GNDs.
- In the unreinforced alloy there is some decrease of the lattice distortion with the heat treatment due to dislocation annihilation and rearrangement. In the composite, instead, the micro RS generated by the GNDs forms back upon cooling, independently of the heat treatment.

APPENDIX

In high-energy synchrotron radiation strain scanning, the gauge volume is a rhomboid prism in which the major diagonal is much longer than the other dimensions of the prism due to the low diffraction angles. Although this limits the spatial resolution in 3-D strain scanning, this length can be optimized by careful considerations on the effective gauge volume.

On ID31 the scattering vector is vertical (see Figs.1.a, 2, A.1). If t is the incident (and diffracted) beam height and b the beam width, the gauge volume can be calculated as the product between the base area and b . If D is the major diagonal and d the minor diagonal:

$$D = \frac{t}{\sin \theta} \quad d = \frac{t}{\cos \theta} \quad (\text{A.1})$$

With these two expressions for the diagonals the total volume can be eventually written as:

$$V_T = \frac{t^2 \cdot b}{\sin 2\theta} \quad (\text{A.2})$$

Let's consider a central portion of D , say X , such that $D = X + Y$. The volume corresponding to this central portion is $V = V_T - V'$, where:

$$V' = \frac{y^2 \tan \theta}{2} b \quad (\text{A.3})$$

is the external volume. From eqs.(A.2-A.3) follows that:

$$V = \frac{t^2 b}{\sin 2\theta} - \frac{(D - X)^2 \tan \theta}{2} b \quad (\text{A.4})$$

If now V and X are normalized to their maximum values ($V = V_T$ and $X = D$, respectively), the following expression is obtained:

$$\frac{V}{V_T} = \frac{X}{D} \left[2 - \frac{X}{D} \right] \quad (\text{A.5})$$

From eq.(A.5) it follows that, if one considers the 50% of the major diagonal, i.e. $X = D/2$, the volume subtended corresponds to the 75% of the total gauge volume, i.e. $V/V_T = 3/4$. This means that it is possible to optimize the spatial resolution needed, knowing that within half of the length of the long diagonal, three quarters of the total diffracting volume are concentrated.

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Figure legend

Fig 1. a) Experimental setup on the instrument. The detector lies behind the secondary slit. The rotation table (ω) is on the left b) Vertical mount c) Horizontal mount d) Geometry and dimensions of the gauge volume

Fig. 2 Geometry of the different components in the axial and radial/hoop mounts.

Fig. 3 ε vs. $\sin^2 \psi$ plots in the central point of samples E219 and E220, in two different heat treatment conditions, T4 and OA (see text).

Fig.4 Macroscopic RS profiles as a function of radius in the three different precipitation conditions in the two materials, E219 and E220.

Fig. 5 Microscopic RS profiles as a function of the radius in the composite E219. Three conditions are shown, a) T4, b) T6, c) Overaged (OA)

Fig. 6 Diffraction-peak width of the Al phase as a function of the sample radius for the composite and the unreinforced alloy

Fig. 7 Relaxation of Macro-RS from T4 to T6 a) alloy E220 b) composite E219

Fig. 8 Relaxation of M-RS from T6 to OA a) E220 b) E219

Fig. 9 Deviatoric and Hydrostatic Micro RS profiles in the composite E219. Three conditions are shown, a) T4, b) T6, c) Overaged (OA)

Fig.A.1- Scheme of the gauge volume. The portion X of the long diagonal identifies the shadowed volume. The beam width b is perpendicular to the plane shown.

Tables

	E219					E220				
	Ax	Rad	Hoop	Hyd	Dev	Ax	Rad	Hoop	Hyd	Dev
T4	146 (10)	92 (15)	146 (15)	128 (14)	18 (18)	195 (2)	81 (2)	204 (2)	160 (2)	35 (4)
T6	42 (10)	36 (20)	18 (15)	33 (16)	9 (22)	53 (2)	17 (2)	26 (2)	32 (2)	21 (4)
OA	4 (10)	0 (20)	0 (15)	1 (16)	4 (22)	18 (2)	0 (2)	0 (2)	6 (2)	14 (4)

Table 1 Macroscopic RS variation (MPa) of the profiles shown in figure 3. $\Delta\sigma = \sigma_{\max} - \sigma_{\min}$.

Errors are shown in brackets.

Condition	Al Phase			SiC Phase		
	Axial	Radial	Hoop	Axial	Radial	Hoop
T4	62 (6)	47 (12)	54 (9)	-334 (50)	-315 (106)	-326 (101)
T6	68 (6)	62 (11)	59 (12)	-388 (60)	-348 (104)	-321 (139)
OA	60 (7)	47 (10)	58 (14)	-357 (71)	-347 (101)	-350 (163)

Table 2 Microscopic RS values (MPa) in E219. Errors are shown in brackets.